

## RAW MATERIALS

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### EFFECT OF COMPLEX THINNING COMPOSITION BASED ON OXYETHYLIDENEDIPHOSPHONIC ACID, LIQUID GLASS, AND SODA ON THE STRUCTURAL–MECHANICAL PROPERTIES OF A CLAY SUSPENSION

G. V. Mozgvoi,<sup>1</sup> M. F. Butman,<sup>1,2</sup> G. P. Kozlovskaya,<sup>1</sup> and E. V. Nikolaeva<sup>1</sup>

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The effect of a complex additive based on oxyethylidenediphosphonic acid, soda, and liquid glass on the viscosity and thixotropy of a Stephan Schmidt clay suspension is studied. The full factorial experiment method is used to obtain regression equations describing the dependence of the structural–mechanical properties of the suspension on the composition and quantity of the complex additive. The compositions of the thinning agent which give the lowest possible viscosity and thixotropy are recommended.

**Key words:** ceramic slip, clay suspension, complex thinning additive, thixotropy, oxyethylidenediphosphonic acid.

The production of ceramic sanitary wares by casting under pressure makes it possible to obtain a strictly prescribed thickness which is the same over all parts of an article. This eliminates many negative factors associated with the appearance of defects on sites with a thickness differential. Article quality largely depends on how well the technological settings of the casting program conform to the slip parameters. For this reason, the key problem of the technology is to make slips whose properties would conform to the rapid structure formation and strength buildup in fired paste. Thus, the study of the rheological stability of slip and finding effective thinners are especially topical problems [1].

The objective of the present work is to control the rheological properties of the ceramic slip used at “Syzranskaya keramika” JSC for casting in polymer molds under high pressure using a combination of components as the thinner: liquid glass, soda, and oxyethylidenediphosphonic acid (OEDPA). The introduction of OEDPA is based on the fact that it has been used effectively for preparing casting slips using the conventional ceramic technology [2, 3].

Casting slip is obtained in three steps: 1) preparation of a Stephan Schmidt clay suspension in a high-speed paddle mixer; 2) preparation of a suspension of inert materials by

grinding in a ball mill; 3) mixing a clay suspension with a suspension of inert materials and with kaolins. The present study has a bearing on the first of the steps indicated above and is associated with the effect of a complex additive on the structural–mechanical properties of a clay suspension. To this end, three suspensions were prepared (content, wt.%<sup>3</sup>): No. 1 — reference suspension with a factory additive (0.500 soda and 0.072 liquid glass); No. 2 — conformance prototype [2] (0.572 soda and 0.572 OEDPK); No. 3 — factory prescription modified with OEDPK additive (0.500 soda, 0.072 liquid glass, and 0.572 OEDPK).

Clays were dissolved in a laboratory high-speed paddle mixer. The moisture content of the clay suspension, taken to be the same for all compositions (35%), was checked by the pycnometric method according to the density of the suspension. The temperature of the suspension was maintained at  $25.0 \pm 0.1^\circ\text{C}$ . The thixotropy was determined according to the difference between the suspension viscosity and viscosity after 1 or 6 min standing. The experimental data, including the dissolution time of the clays, are presented in Table 1.

As Table 1 shows, the suspension No. 2 with complex thinner has unquestionable advantages over a suspension with the factory thinner. The complex thinner in suspension No. 3 has an even stronger effect on a clay suspension.

<sup>1</sup> Ivanovo State Chemical Technology University, Ivanovo, Russia.

<sup>2</sup> E-mail: butman@isuct.ru.

<sup>3</sup> Here and below, content by weight.

**TABLE 1.** Properties of the Suspensions Nos. 1 – 3

Parameter	Check apparatus	Suspension No. 1	Suspension No. 2	Suspension No. 3
Dissolution time, h:min	Timer	6:30	5:00	3:00
Density, g/cm <sup>3</sup> ( $\pm 0.01$ )	Pycnometer, scales	1.61	1.61	1.61
Viscosity, Pa · sec ( $\pm 0.001$ )	Galenkamp viscosimeter	1.009	0.537	0.206
Thixotropy (every 1 min of standing), Pa · sec ( $\pm 0.001$ )	Galenkamp viscosimeter	1.733	0.288	0.000
Thixotropy (every 6 min of standing), Pa · sec ( $\pm 0.001$ )	Galenkamp viscosimeter	6.246	1.492	0.019

Compared with the factory suspension No. 1 it does the following:

- decreases the dissolution time by more than a factor of 2;
- decreases the viscosity by 0.8 Pa · sec;
- decreases the thixotropy to minimal values.

Thus, the first part of these studies showed that, first, a complex thinner [2] based on soda and OEDPK is effective

and, second, it is desirable to replace a portion of the soda with liquid glass.

The second part of these studies consisted of finding the optimal composition of the components of a complex thinner. To accelerate the search process we used the full factorial experiment method (FFE) [4]. The FFE matrix was constructed so that the previous studies performed for suspension No. 3 entered into it as a point with the coordinates +1, +1, +1. A thinner composition containing 0.472% OEDPK, 0.400% soda, and 0.057% liquid glass was chosen as the center of the plan. The ranges of variation of the OEDPK and soda concentrations were  $\Delta x_1 = 0.100\%$  and  $\Delta x_3 = 0.015\%$ , respectively. The FFE plan, including the experiment at the center of the plan, required for checking the adequacy of the regression equation, is presented in Table 2.

Two experiments were performed at each point of the plan. Once again, all studies were performed with the identical moisture content (35%) and density (1.61 g/cm<sup>3</sup>) of the clay suspension and at constant temperature 25°C. The results of the FFE plan implementation are presented in Table 3 as average values of the quantities studied.

The data in Table 3 confirm that the proposed thinner is highly effective in a wide range of OEDPK, soda, and liquid glass concentrations. Eight of the nine suspensions have stable, high structural–mechanical properties which are close in magnitude. The parameters of the suspension No. 8 with the minimum content of OEDPK, soda, and liquid glass (0.372, 0.300, and 0.042%, respectively) are somewhat worse. However, the properties of even this suspension are much higher than for the suspension with the factory additive (No. 1, Table 1).

Analysis of the results of FFE plan implementation (see Table 3) shows that within the limits of the measurement error the variation of the composition and the amount of complex thinner affects the values of the viscosity of the slip as well as the values of the thixotropy of the suspension after 6 min of standing. These indicators were taken into account in the development of the mathematical model used to obtain the regression equations describing the effect of the composition of the thinner on the viscosity and thixotropy of the suspension.

First, the FFE results for the values of the viscosity of the clay suspension were analyzed statistically. The regression

**TABLE 2.** Plan of the Full Factorial Experiment

Experiment No.	Factors in dimensionless coordinates			Factors in a natural scale (thinner content, wt.%)		
	OEDPK $X_1$	Soda $X_2$	Liquid glass $X_3$	OEDPK $x_1$	Soda $x_2$	Liquid glass $x_3$
1	+1	+1	+1	0.572	0.500	0.072
2	–1	+1	+1	0.372	0.500	0.072
3	+1	–1	+1	0.572	0.300	0.072
4	–1	–1	+1	0.372	0.300	0.072
5	+1	+1	–1	0.572	0.500	0.042
6	–1	+1	–1	0.372	0.500	0.042
7	+1	–1	–1	0.572	0.300	0.042
8	–1	–1	–1	0.372	0.300	0.042
Experiment at plan center						
9	0	0	0	0.472	0.400	0.057

**TABLE 3.** Results of Full Factorial Experiment Implementation

Experiment No.	Dissolution time, h:min	Viscosity, Pa · sec	Thixotropy, Pa · sec	
			after 1 min	after 6 min
1	3:00	0.206	0.000	0.019
2	2:30	0.193	0.000	0.007
3	3:40	0.174	0.019	0.055
4	2:40	0.168	0.006	0.019
5	2:50	0.200	0.000	0.000
6	3:00	0.174	0.019	0.068
7	2:40	0.238	0.000	0.008
8	4:30	0.314	0.045	0.154
Experiment at plan center				
9	3:10	0.215	0.012	0.040

equation expressing the dependence of the viscosity  $\eta$  (Pa · sec) of the clay suspension on the quantity and composition of the complex thinner has the form

$$\eta = 0.209 - 0.004X_1 - 0.015X_2 - 0.023X_3 + 0.014X_1X_2 + 0.009X_1X_3 + 0.029X_2X_3 - 0.019X_1X_2X_3. \quad (1)$$

The following equation was obtained from a similar analysis of the dependence of the thixotropy  $T_6$  (Pa · sec) after 6 min of standing of the suspension on the amount and composition of the thinner:

$$T_6 = 0.041 - 0.021X_1 - 0.018X_2 - 0.016X_3 + 0.007X_1X_2 + 0.033X_1X_3 + 0.006X_2X_3 - 0.013X_1X_2X_3. \quad (2)$$

It should be noted that, for all practical purposes, the compositions Nos. 2 and 5 have no thixotropic properties after 6 min of standing (see Table 3). These are the thinner compositions that are proposed for obtaining a clay suspension in the technology of sanitary ware and building articles.

## CONCLUSIONS

The complex thinner OEDP – soda greatly increases the structural–mechanical properties of a Stephan Schmidt clay suspension: the viscosity of the suspension and the thixotropy decrease with decreasing dissolution time.

Adding a small amount of liquid glass to the composition of the complex thinner intensifies the effect of the thinner.

The studies performed using the mathematical planning method established that the thinning effect of the complex additive OEDP – salt – liquid glass is observed in a wide range of concentrations. This makes it possible to confidently regulate the clay dissolution process under production conditions and to obtain suspensions with stable properties.

The optimal compositions (%) of the complex thinner OEDP : soda : liquid glass are proposed on the basis of the implementation of a full factorial experiment: 1) 39.4 : 53.0 : 7.6 (total amount of thinner 0.944%) and 2) 51.3 : 44.9 : 3.8 (total amount of thinner 1.114%).

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